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U.S. ARMY CHEMICAL AND BIOLOGICAL DEFENSE COMMAND

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**ACCELERATED SOLVENT EXTRACTION OF SOIL SAMPLES
FOR THE DETERMINATION OF THE PRESENCE
OF CHEMICAL WARFARE (CW) BREAKDOWN PRODUCTS**

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RESEARCH AND TECHNOLOGY DIRECTORATE

March 1998

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| 14. SUBJECT TERMS Soils Extraction Concentration Chemical agents Breakdown products Thiodiglycol (TDG) Methyl phosphonic acid (MPA) Ethylmethyl phosphonic acid (EMPA) Isopropylmethyl phosphonic acid (IMPA) Pinacolylmethyl phosphonic acid (PMPA) | | | | 15. NUMBER OF PAGES 44 | |
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PREFACE

The work described in this report was authorized in support of the Defense Special Weapons Agency (DSWA) under Project No. DSWA96-2106, Investigate Faster and More Accurate Sample Preparation Procedures, Task 3.4, Dionex Accelerated Solvent Extractor (ASE) 200, and Task 3.6.3, Zymark LV Evaporator. This work was started in August 1996 and completed in January 1997.

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ACCELERATED SOLVENT EXTRACTION OF SOIL SAMPLES FOR THE DETERMINATION OF THE PRESENCE OF CHEMICAL WARFARE (CW) BREAKDOWN PRODUCTS

1. Introduction

Allegations concerning the use and/or manufacture of chemical warfare (CW) agents in recent years has been of interest to many countries.¹ The presence of CW agents, precursors and decomposition (breakdown) products in the environment has been studied by various collection and extraction means.² Solid adsorbent technology has been used to directly adsorb vaporous chemicals in the atmosphere and as a means of concentrating matrixed volatile organic chemicals (VOC's) and semi-volatile organic chemicals (SVOC's).³ Monitoring instrumentation also uses solid adsorbents in demilitarization areas.

Methods of extraction used in the past centered around "shake and settle" technology involving the addition of extraction solvent to a soil, etc. matrix and mixing of the solvent/matrix, followed by removal of the solvent, and concentrating before analyses. Various techniques of solvent separation involved filtration (filter paper), other mechanical means such as vacuum filtration or centrifugation, followed by decantation. Samples were then concentrated by blowing a stream of nitrogen gas across the surface of the liquid in its vessel. Soxhlet extraction was also available, but this involved long extraction times and generated large amounts of solvent which have to be disposed of through time consuming means according to strict requirements involving state and federal laws.

A new approach was sought and found in the use of the Dionex Accelerated Solvent Extraction system (ASE 200)⁴ (figure 1) followed by solvent reduction by the use of a Zymark TurboVap LV Evaporation system (figure 2). The Dionex ASE 200 achieves rapid extraction with organic solvent (alone or as a mixture) at a high temperature under pressures set by the operator on a computerized menu. Thus, solvents remain in the liquid phase by the pressure applied to the cell. The kinetic process of extraction is accelerated. And the analytes of interest are desorbed from the matrix (in this case soil) faster than when compared to room temperature extractions. Low amounts of solvent were generated (typically, 10-18 mL for an 11 mm extraction cell) for a 5 gram sample. The extraction solvents were then reduced on the Zymark LV system that uses a cycloned stream of nitrogen which creates a greater surface area for evaporation in a heated water bath. Parameters on each instrument can be varied to create optimum extraction and concentration conditions.

The work detailed in this report was performed by the Analytical Chemistry Team, Edgewood Research and Technology Directorate, U.S. Army Chemical and Biological Defense Command (CBDCOM) in support of the Defense Special Weapons Agency (DSWA). The objective was to evaluate the performance of the Dionex Accelerated Solvent Extractor (ASE) in the extraction of Chemical Warfare Agents (CWA's) and CW breakdown products from a series of soil matrices (sandy loam, humic and clay). The



Figure 1

Dionex ASE 200 Accelerated Solvent Extractor

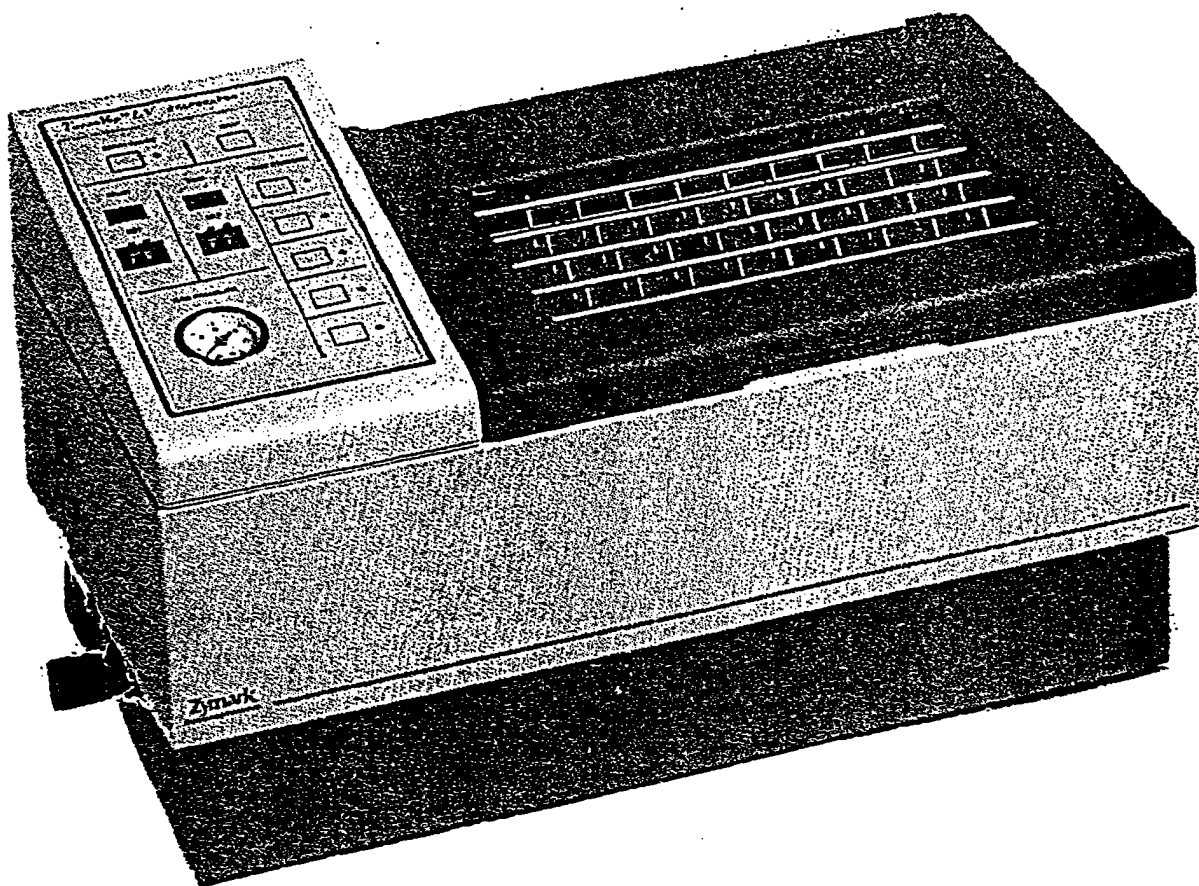


Figure 2

Zymark TurboVap LV Evaporation System

ASE system is seen as a faster, more efficient sample preparation system than that currently used. Different solvent extraction systems were also to be evaluated.

2. Materials and Methods

2.1 Materials

2.1.1 Soils

Soil No. 1 - Sandy Loam - SN 1046, R.T. Corporation - Laramie, WY 82070 (307-7425452)

Soil No. 2 - Sandy Clay-Loam - SN 2002, R.T. Corporation

Soil No. 3 - Loam - SN 3285, R.T. Corporation

(Soils were obtained from R. T. Corporation under contract to Dugway Proving Ground/Defense Special Weapons Agency (DPG/DSWA)).

2.1.2 Sand

Ottawa Sand Standard, 20-30 mesh, Fisher cat no. S23-3 - Fisher Scientific, 585 Alpha Drive, Pittsburgh, PA 15238 (412-963-3300)

2.1.3 Chemicals

MPA (methylphosphonic acid) - ERDEC produced

EMPA (ethylmethylphosphonic acid) - ERDEC produced

IMPA (isopropylmethylphosphonic acid) - ERDEC produced

PMPA (pinacolylmethylphosphonic acid) - ERDEC produced

T-butyl-phosphonic acid (Internal Standard)

TDG (thiodiglycol) - Aldrich (lot # HY 09226CY) - 98% - Aldrich Chemical Co., P.O. Box 355, Milwaukee, WI 53201 (414-273-3850)

Methyl Alcohol GC/MS grade, cat no. 230-4, Baxter (Burdick & Jackson) Muskegon, MI 49442 (616-726-3171)

L-Histidine - Sigma Chemical Company, P.O. Box 14508, St. Louis, MO. (800-325-3010)

2-(N-Morpholino)ethanesulfonic acid (MES) - Sigma Chemical Company, P.O. Box 14508, St. Louis, MO. (800-325-3010)

Tetradecyltrimethylammonium bromide - Sigma Chemical Company, P.O. Box 14508, St. Louis, MO. (800-325-3010)

Triton X-100 - Sigma Chemical Company, P.O. Box 14508, St. Louis, MO. (800-325-3010)

2.2 Instrumentation

Dionex Accelerated Solvent Extractor (ASE 200), (Dionex Corp., 1228 Titan Way, P.O. Box 3603, Sunnyvale CA 94088-3603 (408-737-0700)

Zymark TurboVap LV Concentration Workstation, Zymark Corp., Zymark Center, Hopkinton, MA 01748 (508-435-9500)

THERMO-CE/Crystal Model 300 Capillary Ion Analyzer- Crystal Model 1000 Conductivity Detector, Thermo Capillary Electrophoresis, Inc., Franklin, MA 02038 (508-528-0551)

Waters Millennium 2010 Data Work Station equipped with a Rheodyne Injector, 2 -Model 510 pumps and a Waters Model 490 U.V. Detector, Waters Corporation, Milford, MA 01757-3696 (508-482-3643)

2.3 Extraction Procedure

The Dionex ASE 200 Accelerated Solvent Extractor System in conjunction with the Zymark TurboVap LV Evaporator System were used to prepare previously spiked DPG/DSWA provided soils (Sandy Loam, Sandy Clay-Loam and Loam) for analysis for recoveries of MPA, EMPA, IMPA and PMPA. Samples were processed at 100 degrees C and, in some cases, 150 degrees C to see if enhanced recoveries of the acids from the soil matrices were possible. The operating pressure was maintained at 2000 psig as suggested by the Dionex Corporation. Specifications of each DPG/DSWA standard soil can be viewed as Appendices A, B and C.

Approximately 5 grams of matrix was weighed into a 25 mL bottle with Teflon lined-screw caps for spiking. Aliquots of the dilute standard materials (CW breakdown products in methanol) were added to the matrix. Each soil type was spiked with two levels of chemicals (5 mg and 2.5 mg) and processed in triplicate. Each matrix was allowed to stand for one half hour to evaporate the solvent. The bottles were then each sealed and mixed to distribute the analyte by hand shaking and vortex mixing.

Prior to the addition of the spiked matrix to the Dionex 11 mm extraction cell, a 1.91 cm Whatman filter pad (Whatman cat. no. 10289356) was placed in the bottom of the cell. Two and one half grams of inert Ottawa sand, 20-30 mesh (Fisher cat. no. S23-3) was then added to the cell to provide a barrier to fines from the matrix, which might clog the fine wire mesh screen lining the exit area of the housing of the extraction cell. The spiked sample matrix was then added to the cell, and the cell was tapped to compress the matrix. A second portion of inert sand was added to the cell to within approximately 1 mm of the top edge of the cell. The top (inlet) cap was screwed onto the cell and hand tightened. Each cell was then placed in its appropriate position of the ASE extractor.

After flushing the lines of the ASE 200 four times with solvent (mix), the Method for extraction was entered into the programmer, and the Method was started. Parameters used in the testing of the agent-spiked samples can be seen in Appendix D. After each cell had been processed (approximately 15 minutes), the collection vials were then removed, the extraction volumes measured, and the volume reduced to near dryness using the Zymark TurboVap LV solvent evaporation system. Typical volumes of 20 mLs were reduced. The samples were then reconstituted with one milliliter of methanol each. The reconstituted samples were then analyzed by using a THERMO-CE/Crystal Model 300 Capillary Ion Analyzer - Crystal Model 1000 Conductivity Detector.

The Zymark TurboVap LV system uses a cyclone evaporator system whereby an angular stream of nitrogen against the side of the test tube (in the temperature controlled water bath) causes an increase in the surface area being evaporated per time period. Evaporation times of extracts can be optimized before analyses. Evaporation data charts have been accomplished for individual solvents and solvent mixtures at various temperatures and nitrogen gas flows (pressures). Individual evaporation graphs are included as Appendix E and a table listing the temperatures and pressures for the solvent/solvent mixtures is seen below as Table 3.

TABLE 1
EVAPORATION PARAMETERS
Zymark TurboVap LV

| SOLVENT (S) | TEMP °C | PRESSURE N ₂ PSI | 20--> 2 mL MINUTES |
|--------------------------------|---------|--------------------------------|-----------------------|
| Hexane | 40 | 7 | 30 |
| Hexane | 45 | 7 | 25 |
| Chloroform | 40 | 7 | 40 |
| Chloroform | 45 | 7 | 35 |
| Methanol | 40 | 7 | 80 |
| Methanol | 45 | 7 | 65 |
| Methanol | 50 | 7 | 55 extrapolated |
| Water | 50 | 11 | 300 |
| Methylene chloride | 40 | 7 | 26 |
| Methylene chloride | 45 | 7 | 20 |
| Acetone | 45 | 11 | 20 |
| Methylene chloride/ Acetone | 45 | 11 | 23 extrapolated |

Evaporation curves for Table 1 are provided as Appendix E. An additional chart for methyl alcohol is included as Appendix F ; it shows evaporation times @ 40 degrees C for the three soils.

2.4 Analytical Procedure

The procedure of Rosso and Bossle⁵ was used for analyzing MPA, EMPA, IMPA, and PMPA, and is as follows: electrolyte: 30 mM L-histidine, 30 mM MES, 0.7 mM Tetradecyltrimethylammonium hydroxide (TTAOH) (pH 6.5); 0.03 wt percent Triton X-100; potential -25V; Capillary, CONCAP1TM fused silica; and the capillary temperature = 35 °C. The capillary is regenerated before each analysis with electrolyte for 1.5 minutes. TTAOH is prepared from the bromide salt using a styrene-based anion exchange resin (ONGUARD-A sample pretreatment cartridge, Dionex Corp., Sunnydale, CA).

Analytical Chemistry Team Method 030 (25 August 1997) was used to analyze for TDG and is as follows: column = RP-C18 (150 mm x 4.6 mm); eluent = 25 mM potassium phosphate monobasic (95%)/ acetonitrile (5%); flow rate = 1.0 mL/minute; injection volume = 25uL; and UV wavelength = 225 nm.⁶

3. Discussion

It can be seen in the Table 2A that , although the recoveries of the CWA Breakdown Products are low, the trends are consistent. As we proceed from the Sandy Loam to the Sandy Clay Loam to the Loam matrices, the retention of the breakdown products by the matrix appears to become stronger. The figures suggest that the recovery of MPA from soils is the most difficult, especially from Loam type soil.

Additionally, each soil type was spiked with 5 mg of MPA, IMPA and PMPA and processed at a higher temperature to see whether the processing at a higher temperature might cause more of a release of the breakdown products than that performed at 100 degrees C as seen in Table 2A. The results are seen in Table 2B .

TABLE 2A

EXTRACTION OF SOIL SAMPLES FOR RECOVERY OF CHEMICAL WARFARE BREAKDOWN PRODUCTS -Processed @ 100 degrees C.

| SAMPLE ID. | SOIL TYPE | WEIGHT grams | SPIKE (1 PPT) or (0.5 PPT) MPA, EMPA, IMPA, PMPA with Internal Standard (t-butyl phosphonic acid) | RECOVERED | | | |
|------------|-----------|--------------|--|-----------|---------|---------|---------|
| | | | | MPA | EMPA | IMPA | PMPA |
| RAZ19 | SL | 5.0860 | 5 mg | 2.46 mg | 2.77 mg | 3.74 mg | 3.88 mg |
| RAZ20 | SL | 5.0868 | 5 | 2.33 | 2.86 | 3.96 | 4.23 |
| RAZ21 | SL | 5.0420 | 5 | 2.64 | 2.97 | 4.00 | 4.17 |
| RAZ22 | SL | 5.0912 | 2.5 mg | 1.32 | 2.77 | 2.83 | 2.85 |
| RAZ23 | SL | 5.0163 | 2.5 | 0.903 | 2.52 | 2.64 | 2.66 |
| RAZ24 | SL | 5.0116 | 2.5 | 0.913 | 2.67 | 2.75 | 2.69 |
| RAZ25 | SCL | 4.9653 | 5 mg | 0.577 | 4.26 | 4.00 | 4.56 |
| RAZ26 | SCL | 4.9980 | 5 | 0.683 | 4.00 | 3.92 | 4.47 |
| RAZ27 | SCL | 5.1054 | 5 | 0.680 | 4.20 | 3.91 | 4.44 |
| RAZ28 | SCL | 4.9264 | 2.5 mg | * | 3.06 | 2.84 | 3.38 |
| RAZ29 | SCL | 5.0104 | 2.5 | 0.253 | 2.54 | 2.76 | 3.31 |
| RAZ30 | SCL | 5.0600 | 2.5 | * | 2.73 | 2.96 | 2.88 |
| RAZ31 | L | 4.9739 | 5 mg | * | 1.27 | 2.09 | 3.00 |
| RAZ32 | L | 4.9594 | 5 | * | 1.27 | 2.14 | 3.11 |
| RAZ33 | L | 5.0220 | 5 | * | 1.31 | 2.16 | 3.08 |
| RAZ34 | L | 5.0309 | 2.5 mg | * | 0.650 | 1.04 | 1.89 |
| RAZ35 | L | 5.0823 | 2.5 | * | 0.534 | 0.895 | 1.64 |
| RAZ36 | L | 5.0771 | 2.5 | * | 0.714 | 1.09 | 2.06 |

SL = Sandy Loam
SCL = Sandy Clay Loam

L = Loam
* = below 200 ng per one mL MeOH extract

TABLE 2B

EXTRACTION OF SOIL SAMPLES FOR RECOVERY OF CHEMICAL WARFARE BREAKDOWN
PRODUCTS - Processed @ 150 degrees C.

| SAMPLE ID. | SOIL TYPE | WEIGHT grams | SPIKE (1 PPT) or (0.5 PPT) MPA, EMPA, IMPA, PMPA with Internal Standard (t-butly phosphonic acid) | RECOVERED | | | |
|---------------|--------------|-----------------|--|-----------|---------|--------|--------|
| | | | | MPA | EMPA | IMPA | PMPA |
| RAZ37 | SL | 5.0179 | 5 mg | 1.66 mg | 3.75 mg | 3.89mg | 4.03mg |
| RAZ38 | SL | 5.0053 | 5 | 1.57 | 3.84 | 3.96 | 4.11 |
| RAZ39 | SL | 5.0014 | 5 | 1.71 | 3.80 | 3.87 | 3.74 |
| RAZ40 | SCL | 5.0553 | 5 mg | BDL | 3.23 | 3.35 | 3.62 |
| RAZ41 | SCL | 5.1032 | 5 | BDL | 3.25 | 3.38 | 3.68 |
| RAZ42 | SCL | 5.0313 | 5 | BDL | 2.99 | 3.33 | 3.43 |
| RAZ43 | L | 5.0385 | 5 mg | * | 0.78 | 1.17 | 1.98 |
| RAZ44 | L | 5.0608 | 5 | * | 0.67 | 1.03 | 1.79 |
| RAZ45 | L | 5.0070 | 5 | * | 0.67 | 1.02 | 1.66 |

SL = Sandy Loam

SCL = Sandy Clay Loam

L = Loam

* = below 200 ng per one mL MeOH extract

In reviewing the data above, it appears that the increase in the processing temperature alone results in lower recoveries of Breakdown products

The next task involved the recovery of thiodiglycol (TDG) which had been spiked into DPG/DSWA soils. These soils were processed according to the procedures delineated for "Chemical warfare Breakdown Products - MPA, EMPA, IMPA and PMPA" found on page 2. Each sample (after spiking) was mixed and allowed to stand for one-half hour at room temperature. The matrixed samples were processed at 100 degrees C and reconstituted with methanol as before noted. These reconstituted samples were analyzed on a Waters Millenium 2010 Data Work Station equipped with a Rheodyne Injector, 2 - Model 510 pumps and a Waters Model 490 U.V. Detector. The results are found in Table 3.

TABLE 3

EXTRACTION OF SOIL SAMPLES FOR RECOVERY OF CHEMICAL WARFARE BREAKDOWN PRODUCTS

| SAMPLE ID. | SOIL TYPE | WEIGHT grams | SPIKE (1 PPT) or (0.5 PPT) | TDG RECOVERED @100 degrees C |
|------------|-----------|--------------|-------------------------------|---------------------------------|
| RAZ46 | SL | 5.0080 | 5mg | 3.36 mg |
| RAZ47 | SL | 5.0242 | 5 | 3.87 |
| RAZ48 | SL | 5.0173 | 5 | 3.62 |
| RAZ55 | SL | 5.0117 | 2.5mg | 1.69 |
| RAZ56 | SL | 5.0155 | 2.5 | 1.84 |
| RAZ57 | SL | 5.0027 | 2.5 | 1.80 |
| RAZ49 | SCL | 5.0404 | 5mg | 4.03 |
| RAZ50 | SCL | 5.0111 | 5 | 3.99 |
| RAZ51 | SCL | 5.0041 | 5 | 3.60 |
| RAZ58 | SCL | 5.0742 | 2.5mg | 2.00 |
| RAZ59 | SCL | 5.0970 | 2.5 | 1.90 |
| RAZ60 | SCL | 5.0690 | 2.5 | 1.83 |
| RAZ52 | L | 5.0023 | 5mg | 3.45 |
| RAZ53 | L | 4.9996 | 5 | 4.54 |
| RAZ54 | L | 5.0013 | 5 | 3.56 |
| RAZ61 | L | 5.0507 | 2.5mg | 1.80 |
| RAZ62 | L | 5.0384 | 2.5 | 1.70 |
| RAZ63 | L | 5.0420 | 2.5 | 1.38 |

SL = Sandy Loam

SCL = Sandy Clay Loam

L = Loam

* = below 200 ng per one mL MeOH extract

The recoveries seem consistent from soil to soil, about 70 %. Further work could include determining the lower limits of extraction from the soils and enhanced recoveries through chemical solvent changes, and long term setting times of the TDG/matrix before analysis.

4. Conclusions

The combination of the Dionex ASE (Accelerated Solvent Extractor) and the Zymark TurboVap LV concentrator system provide a systematic alternative to traditional means of processing soil matrixed samples for CW breakdown products. Rapid completion of processing of samples leads to savings in labor, time, and solvent use minimization.

The low recoveries for the "breakdown" products can partially be attributed to complex and simple interactions between minerals and organics.⁷⁻⁹ Weak to strong binding between organics and soils has been noted in the references given above on soils. These interactions can vary between mineral-organic reactions, dipole-dipole attractions, hydrogen bonding, intercalation and chelation.

The previous work of extracting chemical agents from DPG/DSWA standard soils needs to be repeated using the 1/2 hour setting time and three different temperatures (75/100/150 degrees C) in triplicate for the three soils. At least four different concentration levels need to be addressed; sitting times of 1/2 hr, 2 hrs, 1 day, 3 days, weeks, months, etc. need to be addressed. A separate extraction scheme for 2-(diisopropylamino)ethanol and VX needs to be addressed.

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APPENDIX A

Specifications of Standard Sandy Loam Soil Soil No. 1 Inorganics and Physical

METALS ANALYSIS

| Element | MDL | Value |
|---------------------------------|-------|-------|
| Aluminum (Al) | 3 | 1,400 |
| Antimony (Sb) | 1 | ND |
| Arsenic (As) | 0.5 | 0.33 |
| Barium (Ba) | 0.3 | 23.1 |
| Beryllium (Be) | 0.2 | ND |
| Cadmium (Cd) | 0.3 | ND |
| Calcium, soluble (Sat. Paste) | 0.01 | 5.8 |
| Cation Exchange Capacity (CEC) | 0.03 | 2.0 |
| Chromium (Cr) | 1 | 3.3 |
| Cobalt (Co) | 1 | 0.33 |
| Copper (Cu) | 1 | 2.0 |
| Iron (Fe) | 1 | 2,173 |
| Lead (Pb) | 2 | 4.0 |
| Manganese (Mn) | 0.5 | 90.8 |
| Magnesium (Mg) | 0.02 | 0.74 |
| Mercury (Hg) | 0.02 | ND |
| Nickel (Ni) | 1 | 2.3 |
| Potassium, soluble (Sat. Paste) | 0.008 | 0.65 |
| Selenium (Se) | 0.1 | ND |
| Silver (Ag) | 0.5 | ND |
| Sodium, soluble (Sat. Paste) | 0.01 | 0.26 |
| Thallium (Tl) | 1 | ND |
| Vanadium (V) | 0.5 | 3.6 |
| Zinc (Zn) | 1 | 10.0 |

All values given in mg/Kg except Cation Exchange meq/100g, and Calcium, Magnesium, Potassium, and Sodium meq/L

SOIL ANALYSIS

| Parameter | MDL | Value |
|--|-------|--------------|
| Carbon, total (TC) | | 5,617 ug/g |
| Carbonate, total (as CaCO ₃) | 0.01 | 2.5 % |
| Conductivity @ 25° C | 0.001 | 0.67mmhos/cm |
| Exchangeable Acidity | 0.2 | 9.9 meq/100g |
| Organic Matter | 0.01 | 0.48 % |
| pH, Saturated Paste | 0.1 | 7.5 units |
| Solids, Percent | 0.1 | 99.6 % |
| Sulfate, soluble (Water) | 30 | 130 mg/Kg |
| Sulfur, total | 0.01 | ND % |

Texture by Hydrometer

| | | |
|------------------------|---|-------|
| Clay | 1 | 3.3 % |
| Sand | 1 | 93.3% |
| Silt | 1 | 3.3 % |
| Texture Classification | | S. |

WET CHEMISTRY

| Parameter | MDL | Value |
|-----------------------------------|------|-------|
| Cyanide, reactive | 0.03 | ND |
| Nitrate as N, soluble (Water) | 0.5 | 1.2 |
| Nitrogen, ammonia (KCL) | 0.3 | 2.0 |
| Nitrogen, total Kjeldahl | 0.01 | ND |
| Phosphorus, extractable (AB-DTPA) | 1 | 6.7 |
| Phosphorus, total | 0.01 | 0.01 |
| Sulfide, reactive | 0.1 | ND |

All values given in mg/Kg except Nitrogen, total Kjeldahl and Phosphorus, total which are given in %.

Methods

Analysis

All Metals analyzed by SW-846 6010 and 7000 series, 3051 digestion.

| | |
|-----------------------------------|-----------------------------|
| Cation Exchange | USDA No. 60(19) |
| TC | ASTM D4129 |
| Carbonate | ASTM D4129 |
| Conductivity | M120.1 - Meter |
| Exchangeable Acidity | ASA No. 9 9-4.1 |
| Organic Matter | USDA No. 60 Method 24 |
| pH, Saturated Paste | USDA No. 60 (21A) |
| Solids, % | CLPSW390, Part F, D-98 |
| Sulfate, soluble H ₂ O | M375.3-Gravimetric |
| Sulfur, total | ASTM D-4239-85C, LECO Furn. |
| Texture | ASTM D 422 Hydrometer |

Soil Preparation

| | |
|--------------------|-------------------------|
| AB-DPTA Extraction | ASA No. 9,3-5.34 |
| Air Dry at 34 C | USDA No. 1, 1972 |
| Digestion | M3051, HNO ₃ |
| KCL Extraction | ASA No. 9 33-3.22 |
| Sat. Paste Ext. | M2, USDA Handbook 66 |
| Water Ext. | ASA No. 9 10-2.3.2 |

Wet Chemistry

| | |
|-------------------|----------------------------------|
| CN, reactive | Section 8.3 SW-846 & M9012 |
| Nitrate as N | M353.2 |
| KCL | M350.1 |
| N, total Kjeldahl | M351.2-TKN by Block Digester |
| P, extractable | M365.1 Auto Ascorbic Acid digest |
| Sulfide, reactive | Section 8.3 SW-846 & M9030 |

SW-846 Method 8140**Organo-phosphorus pesticides**

| Parameter | MDL (ug/Kg) | Value |
|------------------|-------------|-------|
| Azinphos methyl | 5 | ND |
| Bolstar | 5 | ND |
| Chlorpyrifos | 5 | ND |
| Coumaphos | 5 | ND |
| Demeton | 5 | ND |
| Diazinon | 5 | ND |
| Dichlorvos | 5 | ND |
| Disulfoton | 5 | ND |
| Ethoprop | 5 | ND |
| Fensulfthion | 5 | ND |
| Fenthion | 5 | ND |
| Merphos | 5 | ND |
| Mevinphos | 5 | ND |
| Naled | 5 | ND |
| Parathion methyl | 5 | ND |
| Phorate | 5 | ND |
| Ronnel | 5 | ND |
| Stirophos | 5 | ND |
| Tokuthion | 5 | ND |
| Trichloronate | 5 | ND |

SW-846 Method 8150B**Chlorinated Herbicides**

| Compound | MDL (ug/Kg) | Value |
|--------------|-------------|-------|
| 2,4-D | 20 | ND |
| 2,4-DB | 5 | ND |
| 2,4,5-TP | 5 | ND |
| 2,4,5-T | 5 | ND |
| Dalapon | 5 | ND |
| Dicamba | 20 | ND |
| Dichloroprop | 20 | ND |
| Dinoseb | 20 | ND |
| MCPA | 20 | ND |
| MCPP | 5 | ND |

SW-846 Method 8080A**Chlorinated Pesticides**

| Compound | MDL (ug/Kg) | Value |
|-----------------------|-------------|-------|
| Aldrin | 8 | ND |
| Alpha-BHC | 8 | ND |
| Beta-BHC | 8 | ND |
| Delta-BHC | 8 | ND |
| Gamma-BHC (Lindane) | 8 | ND |
| Chlordane (technical) | 80 | ND |
| 4,4'-DDD | 16 | ND |
| 4,4'-DDE | 16 | ND |
| 4,4'-DDT | 16 | ND |
| Dieldrin | 16 | ND |
| Endosulfan I | 8 | ND |
| Endosulfan II | 16 | ND |
| Endosulfan sulfate | 16 | ND |
| Endrin | 16 | ND |
| Endrin aldehyde | 16 | ND |
| Endrin Ketone | 16 | ND |
| Heptachlor | 8 | ND |
| Heptachlor epoxide | 8 | ND |
| 4,4' -Methoxychlor | 80 | ND |
| Toxaphene | 80 | ND |

SW-846 Method 8270**Semi-Volatile Organics**

| Compound | MDL (ug/Kg) | Value |
|-----------------------------|-------------|-------|
| Phenol | 330 | ND |
| Bis(2-Chloroethyl) Ether | 330 | ND |
| 2-Chlorophenol | 330 | ND |
| 1,3-Dichlorobenzene | 330 | ND |
| 1,4-Dichlorobenzene | 330 | ND |
| 1,2-Dichlorobenzene | 330 | ND |
| 2-Methylphenol | 330 | ND |
| Bis(2-Chloroisopropyl)ether | 330 | ND |
| 4-Methylphenol | 330 | ND |
| N-Nitroso-Di-N-Propylamine | 330 | ND |
| Hexachloroethane | 330 | ND |
| Nitrobenzene | 330 | ND |
| Isophorone | 330 | ND |
| 2-Nitrophenol | 330 | ND |
| 2,4-Dimethylphenol | 1650 | ND |
| Bis(2-Chloroethoxy)methane | 330 | ND |
| 2,4-Dichlorophenol | 330 | ND |
| 1,2,4-Trichlorobenzene | 330 | ND |
| Naphthalene | 330 | ND |
| 4-Chloroaniline | 330 | ND |
| Hexachlorobutadiene | 330 | ND |
| 4-Chloro-3-Methylphenol | 330 | ND |
| 2-Methylnaphthalene | 330 | ND |
| Hexachlorocyclopentadiene | 330 | ND |
| 2,4,6-Trichlorophenol | 330 | ND |
| 2,4,5-Trichlorophenol | 1650 | ND |
| 2-Chloronaphthalene | 330 | ND |
| 2-Nitroaniline | 1650 | ND |
| Dimethyl Phthalate | 330 | ND |
| Acenaphthylene | 330 | ND |
| 3-Nitroaniline | 1650 | ND |
| Acenaphthene | 330 | ND |
| 2,4-Dinitrophenol | 1650 | ND |
| 4-Nitrophenol | 1650 | ND |
| Dibenzofuran | 330 | ND |
| 2,4-Dinitrotoluene | 330 | ND |
| 2,6-Dinitrotoluene | 330 | ND |
| Diethyl Phthalate | 330 | ND |
| 4-Chlorophenyl Phenyl Ether | 330 | ND |
| Fluorene | 330 | ND |
| 4-Nitroaniline | 1650 | ND |
| 4,6-Dinitro-2-Methylphenol | 1650 | ND |
| N-Nitrosodiphenylamine | 330 | ND |
| 4-Bromophenyl Phenyl Ether | 330 | ND |
| Hexachlorobenzene | 330 | ND |
| Pentachlorophenol | 330 | ND |
| Phenanthrene | 330 | ND |
| Anthracene | 330 | ND |
| Carbazole | 330 | ND |
| Di-N-Butyl Phthalate | 330 | ND |
| Fluoranthene | 330 | ND |
| Pyrene | 330 | ND |
| Butyl Benzyl Phthalate | 330 | ND |
| 3,3'-Dichlorobenzidine | 660 | ND |
| Benzo(a)anthracene | 330 | ND |
| Bis(2-Ethylhexyl)phthalate | 330 | ND |
| Chrysene | 330 | ND |
| Di-N-Octylphthalate | 330 | ND |
| Benzo(b)fluoranthene | 330 | ND |
| Benzo(k)fluoranthene | 330 | ND |
| Benzo(a)pyrene | 330 | ND |
| Indeno(1,2,3-c,d)pyrene | 330 | ND |
| Dibenz(a,h)anthracene | 330 | ND |
| Benzo(g,h,i)perylene | 330 | ND |

SW-846 Method 8260

Volatile Organics

| Compound | MDL (ug/Kg) | Value | | |
|---------------------------|-------------|-------|-----------------------------|-------|
| Chloromethane | 10 | ND | Ethylbenzene | 5 ND |
| Bromomethane | 10 | ND | Styrene | 5 ND |
| Vinyl Chloride | 10 | ND | Xylene (total) | 5 ND |
| Chloroethane | 10 | ND | 1,2-Dichlorobenzene | 5 ND |
| Methylene Chloride | 5 | ND | 1,4-Dichlorobenzene | 5 ND |
| Acrylonitrile | 10 | ND | 1,2-Dibromo-3-chloropropane | 5 ND |
| Acetone | 10 | 178 | 1,2-Dibromoethane | 5 ND |
| Carbon Disulfide | 5 | ND | Dichlorodifluoromethane | 5 ND |
| 1,1-Dichloroethene | 5 | ND | Dibromomethane | 5 ND |
| 1,1-Dichloroethane | 5 | ND | Acrolein | 10 ND |
| cis-1,2-Dichloroethene | 5 | ND | 1,1,1,2-Tetrachloroethane | 5 ND |
| trans-1,2-Dichloroethene | 5 | ND | Trichlorofluoromethane | 5 ND |
| Chloroform | 5 | ND | 1,2,3-Trichloropropane | 5 ND |
| 1,2-Dichloroethane | 5 | ND | 2,2-Dichloropropane | 5 ND |
| 2-Butanone | 10 | 8.4 | 1,1 -Dichloropropene | 5 ND |
| 1,1,1 -Trichloroethane | 5 | ND | 1,3-Dichloropropane | 5 ND |
| Carbon Tetrachloride | 5 | ND | Isopropylbenzene | 5 ND |
| Vinyl Acetate | 10 | ND | Bromobenzene | 5 ND |
| Bromodichloromethane | 5 | ND | n-Propylbenzene | 5 ND |
| 1,2-Dichloropropane | 5 | ND | 2-Chlorotoluene | 5 ND |
| cis-1,3-Dichloropropene | 5 | ND | 4-Chlorotoluene | 5 ND |
| Trichloroethene | 5 | ND | 1,3,5-Trimethylbenzene | 5 ND |
| Dibromochloromethane | 5 | ND | tert-Butylbenzene | 5 ND |
| 1,1,2-Trichloroethane | 5 | ND | 1,2,4-Trimethylbenzene | 5 ND |
| Benzene | 5 | ND | sec-Butylbenzene | 5 ND |
| trans-1,3-dichloropropene | 5 | ND | 4-Isopropyltoluene | 5 ND |
| Bromoform | 5 | ND | 1,3-Dichlorobenzene | 5 ND |
| 4-Methyl-2-Pentanone | 10 | ND | n-Butylbenzene | 5 ND |
| 2-Hexanone | 10 | ND | 1,2,4-Trichlorobenzene | 5 ND |
| Tetrachloroethene | 5 | ND | Hexachlorobutadiene | 5 ND |
| 1,1,2,2-Tetrachloroethane | 5 | ND | Naphthalene | 5 ND |
| Toluene | 5 | 9.7 | 1,2,3-Trichlorobenzene | 5 ND |
| Chlorobenzene | 5 | ND | | |

Blank

APPENDIX B

Specifications of Sandy Clay Loam Soil Soil No. 2 Inorganics and Physical

METALS ANALYSIS

| <u>Element</u> | <u>MDL</u> | <u>Value</u> |
|---------------------------------|------------|--------------|
| Aluminum (Al) | 3 | 3,540 |
| Antimony (Sb) | 1 | ND |
| Arsenic (As) | 0.5 | 2.0 |
| Barium (Ba) | 0.3 | 50.0 |
| Beryllium (Be) | 0.2 | ND |
| Cadmium (Cd) | 0.3 | ND |
| Calcium, soluble (Sat. Paste) | 0.01 | 20.5 |
| Cation Exchange Capacity (CEC) | 0.03 | 13.9 |
| Chromium (Cr) | 1 | ND |
| Cobalt (Co) | 1 | ND |
| Copper (Cu) | 1 | ND |
| Iron (Fe) | 1 | 3,163 |
| Lead (Pb) | 2 | ND |
| Manganese (Mn) | 0.5 | 97.3 |
| Magnesium (Mg) | 0.02 | 228.0 |
| Mercury (Hg) | 0.02 | 0.03 |
| Nickel (Ni) | 1 | ND |
| Potassium, soluble (Sat. Paste) | 0.008 | 5.72 |
| Selenium (Se) | 0.1 | 0.40 |
| Silver (Ag) | 0.5 | ND |
| Sodium, soluble (Sat. Paste) | 0.01 | 38.7 |
| Thallium (Tl) | 1 | ND |
| Vanadium (V) | 0.5 | 16.0 |
| Zinc (Zn) | 1 | 21.3 |

All values given in mg/Kg except Cation Exchange meq/100g, and Calcium, Magnesium, Potassium, and Sodium meq/L

SOIL ANALYSIS

| <u>Parameter</u> | <u>MDL</u> | <u>Value</u> |
|--|------------|---------------|
| Carbon, total (TC) | | 1,773 ug/g |
| Carbonate, total (as CaCO ₃) | 0.01 | 4.6 % |
| Conductivity @ 25° C | 0.001 | 14.9mmhos/cm |
| Exchangeable Acidity | 0.2 | 11.3 meq/100g |
| Organic Matter | 0.01 | 1.85 % |
| pH, Saturated Paste | 0.1 | 8.4 units |
| Solids, Percent | 0.1 | 86.0 % |
| Sulfate, soluble (Water) | 30 | 16,700 mg/Kg |
| Sulfur, total | 0.01 | 12.7 % |

Texture by Hydrometer

| | | |
|------------------------|---|--------|
| Clay | 1 | 10.0 % |
| Sand | 1 | 50.0 % |
| Silt | 1 | 40.0 % |
| Texture Classification | | L |

WET CHEMISTRY

| <u>Parameter</u> | <u>MDL</u> | <u>Value</u> |
|-----------------------------------|------------|--------------|
| Cyanide, reactive | 0.03 | ND |
| Nitrate as N, soluble (Water) | 0.5 | 34.3 |
| Nitrogen, ammonia (KCL) | 0.3 | 4.7 |
| Nitrogen, total Kjeldahl | 0.01 | 0.13 |
| Phosphorus, extractable (AB-DTPA) | 1 | 1.0 |
| Phosphorus, total | 0.01 | 0.02 |
| Sulfide, reactive | 0.1 | ND |

All values given in mg/Kg except Nitrogen, total Kjeldahl and Phosphorus, total which are given in %.

Methods

Analysis

| | |
|---|-----------------------------|
| All Metals analyzed by SW-846 6010 and 7000 series, 3051 digestion. | |
| Cation Exchange | USDA No. 60(19) |
| TC | ASTM D4129 |
| Carbonate | ASTM D4129 |
| Conductivity | M120.1 - Meter |
| Exchangeable Acidity | ASA No. 9 9-4.1 |
| Organic Matter | USDA No. 60 Method 24 |
| pH, Saturated Paste | USDA No. 60 (21A) |
| Solids, % | CLPSW390, Part F, D-98 |
| Sulfate, soluble H ₂ O | M375.3-Gravimetric |
| Sulfur, total | ASTM D-4239-85C, LECO Furn. |
| Texture | ASTM D 422 Hydrometer |

Soil Preparation

| | |
|--------------------|-------------------------|
| AB-DPTA Extraction | ASA No. 9,3-5.34 |
| Air Dry at 34 C | USDA No. 1, 1972 |
| Digestion | M3051, HNO ₃ |
| KCL Extraction | ASA No. 9 33-3.22 |
| Sat. Paste Ext. | M2, USDA Handbook 66 |
| Water Ext. | ASA No. 9 10-2.3.2 |

Wet Chemistry

| | |
|-------------------|----------------------------------|
| CN, reactive | Section 8.3 SW-846 & M9012 |
| Nitrate as N | M353.2 |
| KCL | M350.1 |
| N, total Kjeldahl | M351.2-TKN by Block Digester |
| P, extractable | M365.1 Auto Ascorbic Acid digest |
| Sulfide, reactive | Section 8.3 SW-846 & M9030 |

SW-846 Method 8140

Organo-phosphorus pesticides

| Parameter | MDL (ug/Kg) | Value |
|------------------|-------------|-------|
| Azinphos methyl | 5 | ND |
| Bolstar | 5 | ND |
| Chlorpyrifos | 5 | ND |
| Coumaphos | 5 | ND |
| Demeton | 5 | ND |
| Diazinon | 5 | ND |
| Dichlorvos | 5 | ND |
| Disulfoton | 5 | ND |
| Ethoprop | 5 | ND |
| Fensulfothion | 5 | ND |
| Fenthion | 5 | ND |
| Merphos | 5 | ND |
| Mevinphos | 5 | ND |
| Naled | 5 | ND |
| Parathion methyl | 5 | ND |
| Phorate | 5 | ND |
| Ronnel | 5 | ND |
| Stirophos | 5 | ND |
| Tokuthion | 5 | ND |
| Trichloronate | 5 | ND |

SW-846 Method 8150B

Chlorinated Herbicides

| Compound | MDL (ug/Kg) | Value |
|--------------|-------------|-------|
| 2,4-D | 20 | ND |
| 2,4-DB | 5 | ND |
| 2,4,5-TP | 5 | ND |
| 2,4,5-T | 5 | ND |
| Dalapon | 5 | ND |
| Dicamba | 20 | ND |
| Dichloroprop | 20 | ND |
| Dinoseb | 20 | ND |
| MCPA | 20 | ND |
| MCPP | 5 | ND |

SW-846 Method 8080A

Chlorinated Pesticides

| Compound | MDL (ug/Kg) | Value |
|-----------------------|-------------|-------|
| Aldrin | 8 | ND |
| Alpha-BHC | 8 | ND |
| Beta-BHC | 8 | ND |
| Delta-BHC | 8 | ND |
| Gamma-BHC (Lindane) | 8 | ND |
| Chlordane (technical) | 80 | ND |
| 4,4'-DDD | 16 | ND |
| 4,4'-DDE | 16 | ND |
| 4,4'-DDT | 16 | ND |
| Dieldrin | 16 | ND |
| Endosulfan I | 8 | ND |
| Endosulfan II | 16 | ND |
| Endosulfan sulfate | 16 | ND |
| Endrin | 16 | ND |
| Endrin aldehyde | 16 | ND |
| Endrin Ketone | 16 | ND |
| Heptachlor | 8 | ND |
| Heptachlor epoxide | 8 | ND |
| 4,4' -Methoxychlor | 80 | ND |
| Toxaphene | 80 | ND |

SW-846 Method 8270

Semi-Volatile Organics

| Compound | MDL (ug/Kg) | Value |
|-----------------------------|-------------|-------|
| Phenol | 330 | ND |
| Bis(2-Chloroethyl) Ether | 330 | ND |
| 2-Chlorophenol | 330 | ND |
| 1,3-Dichlorobenzene | 330 | ND |
| 1,4-Dichlorobenzene | 330 | ND |
| 1,2-Dichlorobenzene | 330 | ND |
| 2-Methylphenol | 330 | ND |
| Bis(2-Chloroisopropyl)ether | 330 | ND |
| 4-Methylphenol | 330 | ND |
| N-Nitroso-Di-N-Propylamine | 330 | ND |
| Hexachloroethane | 330 | ND |
| Nitrobenzene | 330 | ND |
| Isophorone | 330 | ND |
| 2-Nitrophenol | 330 | ND |
| 2,4-Dimethylphenol | 1650 | ND |
| Bis(2-Chloroethoxy)methane | 330 | ND |
| 2,4-Dichlorophenol | 330 | ND |
| 1,2,4-Trichlorobenzene | 330 | ND |
| Naphthalene | 330 | ND |
| 4-Chloroaniline | 330 | ND |
| Hexachlorobutadiene | 330 | ND |
| 4-Chloro-3-Methylphenol | 330 | ND |
| 2-Methylnaphthalene | 330 | ND |
| Hexachlorocyclopentadiene | 330 | ND |
| 2,4,6-Trichlorophenol | 330 | ND |
| 2,4,5-Trichlorophenol | 1650 | ND |
| 2-Chloronaphthalene | 330 | ND |
| 2-Nitroaniline | 1650 | ND |
| Dimethyl Phthalate | 330 | ND |
| Acenaphthylene | 330 | ND |
| 3-Nitroaniline | 1650 | ND |
| Acenaphthene | 330 | ND |
| 2,4-Dinitrophenol | 1650 | ND |
| 4-Nitrophenol | 1650 | ND |
| Dibenzofuran | 330 | ND |
| 2,4-Dinitrotoluene | 330 | ND |
| 2,6-Dinitrotoluene | 330 | ND |
| Diethyl Phthalate | 330 | ND |
| 4-Chlorophenyl Phenyl Ether | 330 | ND |
| Fluorene | 330 | ND |
| 4-Nitroaniline | 1650 | ND |
| 4,6-Dinitro-2-Methylphenol | 1650 | ND |
| N-Nitrosodiphenylamine | 330 | ND |
| 4-Bromophenyl Phenyl Ether | 330 | ND |
| Hexachlorobenzene | 330 | ND |
| Pentachlorophenol | 330 | ND |
| Phenanthrene | 330 | ND |
| Anthracene | 330 | ND |
| Carbazole | 330 | ND |
| Di-N-Butyl Phthalate | 330 | ND |
| Fluoranthene | 330 | ND |
| Pyrene | 330 | ND |
| Butyl Benzyl Phthalate | 330 | ND |
| 3,3'-Dichlorobenzidine | 660 | ND |
| Benzo(a)anthracene | 330 | ND |
| Bis(2-Ethylhexyl)phthalate | 330 | ND |
| Chrysene | 330 | ND |
| Di-N-Octylphthalate | 330 | ND |
| Benzo(b)fluoranthene | 330 | ND |
| Benzo(k)fluoranthene | 330 | ND |
| Benzo(a)pyrene | 330 | ND |
| Indeno(1,2,3-c,d)pyrene | 330 | ND |
| Dibenz(a,h)anthracene | 330 | ND |
| Benzo(g,h,i)perylene | 330 | ND |

SW-846 Method 8260

Volatile Organics

| Compound | MDL (ug/Kg) | Value |
|---------------------------|-------------|-------|
| Chloromethane | 10 | ND |
| Bromomethane | 10 | ND |
| Vinyl Chloride | 10 | ND |
| Chloroethane | 10 | ND |
| Methylene Chloride | 5 | ND |
| Acrylonitrile | 10 | ND |
| Acetone | 10 | 39 |
| Carbon Disulfide | 5 | ND |
| 1,1-Dichloroethene | 5 | ND |
| 1,1-Dichloroethane | 5 | ND |
| cis-1,2-Dichloroethene | 5 | ND |
| trans-1,2-Dichloroethene | 5 | ND |
| Chloroform | 5 | ND |
| 1,2-Dichloroethane | 5 | ND |
| 2-Butanone | 10 | ND |
| 1,1,1-Trichloroethane | 5 | ND |
| Carbon Tetrachloride | 5 | ND |
| Vinyl Acetate | 10 | ND |
| Bromodichloromethane | 5 | ND |
| 1,2-Dichloropropane | 5 | ND |
| cis-1,3-Dichloropropene | 5 | ND |
| Trichloroethene | 5 | ND |
| Dibromochloromethane | 5 | ND |
| 1,1,2-Trichloroethane | 5 | ND |
| Benzene | 5 | ND |
| trans-1,3-dichloropropene | 5 | ND |
| Bromoform | 5 | ND |
| 4-Methyl-2-Pentanone | 10 | ND |
| 2-Hexanone | 10 | ND |
| Tetrachloroethene | 5 | ND |
| 1,1,2,2-Tetrachloroethane | 5 | ND |
| Toluene | 5 | ND |
| Chlorobenzene | 5 | ND |

| | | |
|-----------------------------|----|----|
| Ethylbenzene | 5 | ND |
| Styrene | 5 | ND |
| Xylene (total) | 5 | ND |
| 1,2-Dichlorobenzene | 5 | ND |
| 1,4-Dichlorobenzene | 5 | ND |
| 1,2-Dibromo-3-chloropropane | 5 | ND |
| 1,2-Dibromoethane | 5 | ND |
| Dichlorodifluoromethane | 5 | ND |
| Dibromomethane | 5 | ND |
| Acrolein | 10 | ND |
| 1,1,1,2-Tetrachloroethane | 5 | ND |
| Trichlorofluoromethane | 5 | ND |
| 1,2,3-Trichloropropane | 5 | ND |
| 2,2-Dichloropropane | 5 | ND |
| 1,1-Dichloropropene | 5 | ND |
| 1,3-Dichloropropane | 5 | ND |
| Isopropylbenzene | 5 | ND |
| Bromobenzene | 5 | ND |
| n-Propylbenzene | 5 | ND |
| 2-Chlorotoluene | 5 | ND |
| 4-Chlorotoluene | 5 | ND |
| 1,3,5-Trimethylbenzene | 5 | ND |
| tert-Butylbenzene | 5 | ND |
| 1,2,4-Trimethylbenzene | 5 | ND |
| sec-Butylbenzene | 5 | ND |
| 4-Isopropyltoluene | 5 | ND |
| 1,3-Dichlorobenzene | 5 | ND |
| n-Butylbenzene | 5 | ND |
| 1,2,4-Trichlorobenzene | 5 | ND |
| Hexachlorobutadiene | 5 | ND |
| Naphthalene | 5 | ND |
| 1,2,3-Trichlorobenzene | 5 | ND |

Blank

APPENDIX C

Specifications of Standard Loam Soil Soil No. 3 Inorganics and Physical

METALS ANALYSIS

| Element | MDL | Value |
|---------------------------------|-------|--------|
| Aluminum (Al) | 3 | 11,033 |
| Antimony (Sb) | 1 | ND |
| Arsenic (As) | 0.5 | 2.0 |
| Barium (Ba) | 0.3 | 204.3 |
| Beryllium (Be) | 0.2 | 0.70 |
| Cadmium (Cd) | 0.3 | 0.53 |
| Calcium, soluble (Sat. Paste) | 0.01 | 11.0 |
| Cation Exchange Capacity (CEC) | 0.03 | 14.7 |
| Chromium (Cr) | 1 | 9.0 |
| Cobalt (Co) | 1 | 9.3 |
| Copper (Cu) | 1 | 9.7 |
| Iron (Fe) | 1 | 27,000 |
| Lead (Pb) | 2 | 17.3 |
| Manganese (Mn) | 0.5 | 577.3 |
| Magnesium (Mg) | 0.02 | 3.04 |
| Mercury (Hg) | 0.02 | 0.31 |
| Nickel (Ni) | 1 | 8.0 |
| Potassium, soluble (Sat. Paste) | 0.008 | 0.92 |
| Selenium (Se) | 0.1 | 0.30 |
| Silver (Ag) | 0.5 | ND |
| Sodium, soluble (Sat. Paste) | 0.01 | 0.48 |
| Thallium (Tl) | 1 | ND |
| Vanadium (V) | 0.5 | 17.4 |
| Zinc (Zn) | 1 | 112.3 |

All values given in mg/Kg except Cation Exchange meq/100g, and Calcium, Magnesium, Potassium, and Sodium meq/L

SOIL ANALYSIS

| Parameter | MDL | Value |
|--|-------|---------------|
| Carbon, total (TC) | | 45,067 ug/g |
| Carbonate, total (as CaCO ₃) | 0.01 | 0.04 % |
| Conductivity @ 25° C | 0.001 | 1.27mmhos/cm |
| Exchangeable Acidity | 0.2 | 19.1 meq/100g |
| Organic Matter | 0.01 | 5.96 % |
| pH, Saturated Paste | 0.1 | 5.8 units |
| Solids, Percent | 0.1 | 96.4 % |
| Sulfate, soluble (Water) | 30 | 526.7 mg/Kg |
| Sulfur, total | 0.01 | 0.02 % |

Texture by Hydrometer

| | | |
|------------------------|---|--------|
| Clay | 1 | 10.0 % |
| Sand | 1 | 65.0 % |
| Silt | 1 | 25.0 % |
| Texture Classification | | SL |

WET CHEMISTRY

| Parameter | MDL | Value |
|-----------------------------------|------|-------|
| Cyanide, reactive | 0.03 | ND |
| Nitrate as N, soluble (Water) | 0.5 | 0.57 |
| Nitrogen, ammonia (KCL) | 0.3 | 5.17 |
| Nitrogen, total Kjeldahl | 0.01 | 0.19 |
| Phosphorus, extractable (AB-DTPA) | 1 | 14.3 |
| Phosphorus, total | 0.01 | 0.09 |
| Sulfide, reactive | 0.1 | ND |

All values given in mg/Kg except Nitrogen, total Kjeldahl and Phosphorus, total which are given in %.

Methods

Analysis

All Metals analyzed by SW-846 6010 and 7000 series, 3051 digestion.

| | |
|-----------------------------------|-----------------------------|
| Cation Exchange | USDA No. 60(19) |
| TC | ASTM D4129 |
| Carbonate | ASTM D4129 |
| Conductivity | M120.1 - Meter |
| Exchangeable Acidity | ASA No. 9 9-4.1 |
| Organic Matter | USDA No. 60 Method 24 |
| pH, Saturated Paste | USDA No. 60 (21A) |
| Solids, % | CLPSW390, Part F, D-98 |
| Sulfate, soluble H ₂ O | M375.3-Gravimetric |
| Sulfur, total | ASTM D-4239-85C, LECO Furn. |
| Texture | ASTM D 422 Hydrometer |

Soil Preparation

| | |
|--------------------|-------------------------|
| AB-DPTA Extraction | ASA No. 9,3-5.34 |
| Air Dry at 34 C | USDA No. 1, 1972 |
| Digestion | M3051, HNO ₃ |
| KCL Extraction | ASA No. 9 33-3.22 |
| Sat. Paste Ext. | M2, USDA Handbook 66 |
| Water Ext. | ASA No. 9 10-2.3.2 |

Wet Chemistry

| | |
|-------------------|----------------------------------|
| CN, reactive | Section 8.3 SW-846 & M9012 |
| Nitrate as N | M353.2 |
| KCL | M350.1 |
| N, total Kjeldahl | M351.2-TKN by Block Digester |
| P, extractable | M365.1 Auto Ascorbic Acid digest |
| Sulfide, reactive | Section 8.3 SW-846 & M9030 |

SW-846 Method 8140**Organo-phosphorus pesticides**

| Parameter | MDL (ug/Kg) | Value |
|------------------|-------------|-------|
| Azinphos methyl | 5 | ND |
| Bolstar | 5 | ND |
| Chlorpyrifos | 5 | ND |
| Coumaphos | 5 | ND |
| Demeton | 5 | ND |
| Diazinon | 5 | ND |
| Dichlorvos | 5 | ND |
| Disulfoton | 5 | ND |
| Ethoprop | 5 | ND |
| Fensulfothion | 5 | ND |
| Fenthion | 5 | ND |
| Merphos | 5 | ND |
| Mevinphos | 5 | ND |
| Naled | 5 | ND |
| Parathion methyl | 5 | ND |
| Phorate | 5 | ND |
| Ronnel | 5 | ND |
| Stirophos | 5 | ND |
| Tokuthion | 5 | ND |
| Trichloronate | 5 | ND |

SW-846 Method 8150B**Chlorinated Herbicides**

| Compound | MDL (ug/Kg) | Value |
|--------------|-------------|-------|
| 2,4-D | 20 | ND |
| 2,4-DB | 5 | ND |
| 2,4,5-TP | 5 | ND |
| 2,4,5-T | 5 | ND |
| Dalapon | 5 | ND |
| Dicamba | 20 | ND |
| Dichloroprop | 20 | ND |
| Dinoseb | 20 | ND |
| MCPA | 20 | ND |
| MCPP | 5 | ND |

SW-846 Method 8080A**Chlorinated Pesticides**

| Compound | MDL (ug/Kg) | Value |
|-----------------------|-------------|-------|
| Aldrin | 8 | ND |
| Alpha-BHC | 8 | ND |
| Beta-BHC | 8 | ND |
| Delta-BHC | 8 | ND |
| Gamma-BHC (Lindane) | 8 | ND |
| Chlordane (technical) | 80 | ND |
| 4,4'-DDD | 16 | ND |
| 4,4'-DDE | 16 | ND |
| 4,4'-DDT | 16 | ND |
| Dieldrin | 16 | ND |
| Endosulfan I | 8 | ND |
| Endosulfan II | 16 | ND |
| Endosulfan sulfate | 16 | ND |
| Endrin | 16 | ND |
| Endrin aldehyde | 16 | ND |
| Endrin Ketone | 16 | ND |
| Heptachlor | 8 | ND |
| Heptachlor epoxide | 8 | ND |
| 4,4' -Methoxychlor | 80 | ND |
| Toxaphene | 80 | ND |

SW-846 Method 8270**Semi-Volatile Organics**

| Compound | MDL (ug/Kg) | Value |
|-----------------------------|-------------|-------|
| Phenol | 330 | ND |
| Bis(2-Chloroethyl) Ether | 330 | ND |
| 2-Chlorophenol | 330 | ND |
| 1,3-Dichlorobenzene | 330 | ND |
| 1,4-Dichlorobenzene | 330 | ND |
| 1,2-Dichlorobenzene | 330 | ND |
| 2-Methylphenol | 330 | ND |
| Bis(2-Chloroisopropyl)ether | 330 | ND |
| 4-Methylphenol | 330 | ND |
| N-Nitroso-Di-N-Propylamine | 330 | ND |
| Hexachloroethane | 330 | ND |
| Nitrobenzene | 330 | ND |
| Isophorone | 330 | ND |
| 2-Nitrophenol | 330 | ND |
| 2,4-Dimethylphenol | 1650 | ND |
| Bis(2-Chloroethoxy)methane | 330 | ND |
| 2,4-Dichlorophenol | 330 | ND |
| 1,2,4-Trichlorobenzene | 330 | ND |
| Naphthalene | 330 | ND |
| 4-Chloroaniline | 330 | ND |
| Hexachlorobutadiene | 330 | ND |
| 4-Chloro-3-Methylphenol | 330 | ND |
| 2-Methylnaphthalene | 330 | ND |
| Hexachlorocyclopentadiene | 330 | ND |
| 2,4,6-Trichlorophenol | 330 | ND |
| 2,4,5-Trichlorophenol | 1650 | ND |
| 2-Chloronaphthalene | 330 | ND |
| 2-Nitroaniline | 1650 | ND |
| Dimethyl Phthalate | 330 | ND |
| Acenaphthylene | 330 | ND |
| 3-Nitroaniline | 1650 | ND |
| Acenaphthene | 330 | ND |
| 2,4-Dinitrophenol | 1650 | ND |
| 4-Nitrophenol | 1650 | ND |
| Dibenzofuran | 330 | ND |
| 2,4-Dinitrotoluene | 330 | ND |
| 2,6-Dinitrotoluene | 330 | ND |
| Diethyl Phthalate | 330 | ND |
| 4-Chlorophenyl Phenyl Ether | 330 | ND |
| Fluorene | 330 | ND |
| 4-Nitroaniline | 1650 | ND |
| 4,6-Dinitro-2-Methylphenol | 1650 | ND |
| N-Nitrosodiphenylamine | 330 | ND |
| 4-Bromophenyl Phenyl Ether | 330 | ND |
| Hexachlorobenzene | 330 | ND |
| Pentachlorophenol | 330 | ND |
| Phenanthrene | 330 | ND |
| Anthracene | 330 | ND |
| Carbazole | 330 | ND |
| Di-N-Butyl Phthalate | 330 | ND |
| Fluoranthene | 330 | ND |
| Pyrene | 330 | ND |
| Butyl Benzyl Phthalate | 330 | ND |
| 3,3'-Dichlorobenzidine | 660 | ND |
| Benzo(a)anthracene | 330 | ND |
| Bis(2-Ethylhexyl)phthalate | 330 | ND |
| Chrysene | 330 | ND |
| Di-N-Octylphthalate | 330 | ND |
| Benzo(b)fluoranthene | 330 | ND |
| Benzo(k)fluoranthene | 330 | ND |
| Benzo(a)pyrene | 330 | ND |
| Indeno(1,2,3-c,d)pyrene | 330 | ND |
| Dibenz(a,h)anthracene | 330 | ND |
| Benzo(g,h,i)perylene | 330 | ND |

SW-846 Method 8260

Volatile Organics

| Compound | MDL (ug/Kg) | Value |
|---------------------------|-------------|-------|
| Chloromethane | 10 | ND |
| Bromomethane | 10 | ND |
| Vinyl Chloride | 10 | ND |
| Chloroethane | 10 | ND |
| Methylene Chloride | 5 | ND |
| Acrylonitrile | 10 | ND |
| Acetone | 10 | 38.7 |
| Carbon Disulfide | 5 | ND |
| 1,1-Dichloroethene | 5 | ND |
| 1,1-Dichloroethane | 5 | ND |
| cis-1,2-Dichloroethene | 5 | ND |
| trans-1,2-Dichloroethene | 5 | ND |
| Chloroform | 5 | ND |
| 1,2-Dichloroethane | 5 | ND |
| 2-Butanone | 10 | ND |
| 1,1,1-Trichloroethane | 5 | ND |
| Carbon Tetrachloride | 5 | ND |
| Vinyl Acetate | 10 | ND |
| Bromodichloromethane | 5 | ND |
| 1,2-Dichloropropane | 5 | ND |
| cis-1,3-Dichloropropene | 5 | ND |
| Trichloroethene | 5 | ND |
| Dibromochloromethane | 5 | ND |
| 1,1,2-Trichloroethane | 5 | ND |
| Benzene | 5 | ND |
| trans-1,3-dichloropropene | 5 | ND |
| Bromoform | 5 | ND |
| 4-Methyl-2-Pentanone | 10 | ND |
| 2-Hexanone | 10 | ND |
| Tetrachloroethene | 5 | ND |
| 1,1,2,2-Tetrachloroethane | 5 | ND |
| Toluene | 5 | 13.7 |
| Chlorobenzene | 5 | ND |

| | | |
|-----------------------------|----|------|
| Ethylbenzene | 5 | ND |
| Styrene | 5 | ND |
| Xylene (total) | 5 | 14.3 |
| 1,2-Dichlorobenzene | 5 | ND |
| 1,4-Dichlorobenzene | 5 | ND |
| 1,2-Dibromo-3-chloropropane | 5 | ND |
| 1,2-Dibromoethane | 5 | ND |
| Dichlorodifluoromethane | 5 | ND |
| Dibromomethane | 5 | ND |
| Acrolein | 10 | ND |
| 1,1,1,2-Tetrachloroethane | 5 | ND |
| Trichlorofluoromethane | 5 | ND |
| 1,2,3-Trichloropropane | 5 | ND |
| 2,2-Dichloropropane | 5 | ND |
| 1,1-Dichloropropene | 5 | ND |
| 1,3-Dichloropropane | 5 | ND |
| Isopropylbenzene | 5 | ND |
| Bromobenzene | 5 | ND |
| n-Propylbenzene | 5 | ND |
| 2-Chlorotoluene | 5 | ND |
| 4-Chlorotoluene | 5 | ND |
| 1,3,5-Trimethylbenzene | 5 | ND |
| tert-Butylbenzene | 5 | ND |
| 1,2,4-Trimethylbenzene | 5 | ND |
| sec-Butylbenzene | 5 | ND |
| 4-Isopropyltoluene | 5 | ND |
| 1,3-Dichlorobenzene | 5 | ND |
| n-Butylbenzene | 5 | ND |
| 1,2,4-Trichlorobenzene | 5 | ND |
| Hexachlorobutadiene | 5 | ND |
| Naphthalene | 5 | ND |
| 1,2,3-Trichlorobenzene | 5 | ND |

Blank

APPENDIX D

Dionex ASE 200 Method "for extraction"

DIONEX ACCELERATED SOLVENT EXTRACTOR ASE 200 PARAMETERS

| SOIL TYPE | SANDY LOAM | SANDY CLAY LOAM | LOAM |
|----------------|------------|-----------------|----------|
| HEAT minutes | 5 | 5 | 5 |
| STATIC minutes | 5 | 5 | 5 |
| FLUSH % Volume | 60 | 60 | 60 |
| PURGE seconds | 60 | 60 | 60 |
| CYCLES | 1 | 1 | 1 |
| PRESSURE psi | 2000 | 2000 | 2000 |
| TEMPERATURE °C | 100/150 | 100/150 | 100/150 |
| SOLVENT | Methanol | Methanol | Methanol |
| | | | |

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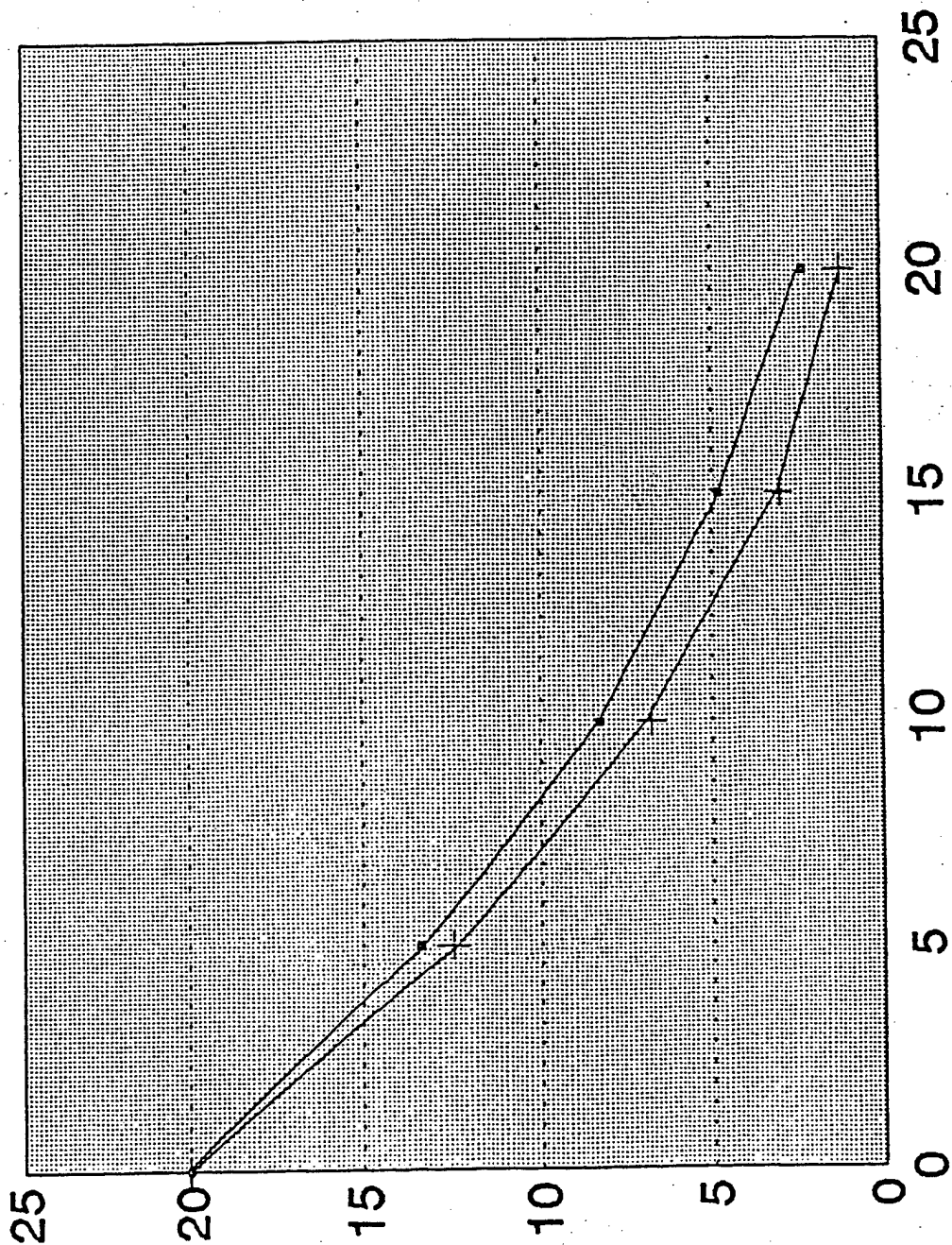
APPENDIX E

Zymark TurboVap LV Evaporation Graphs - Solvents

EVAPORATION DATA

ZYMARK

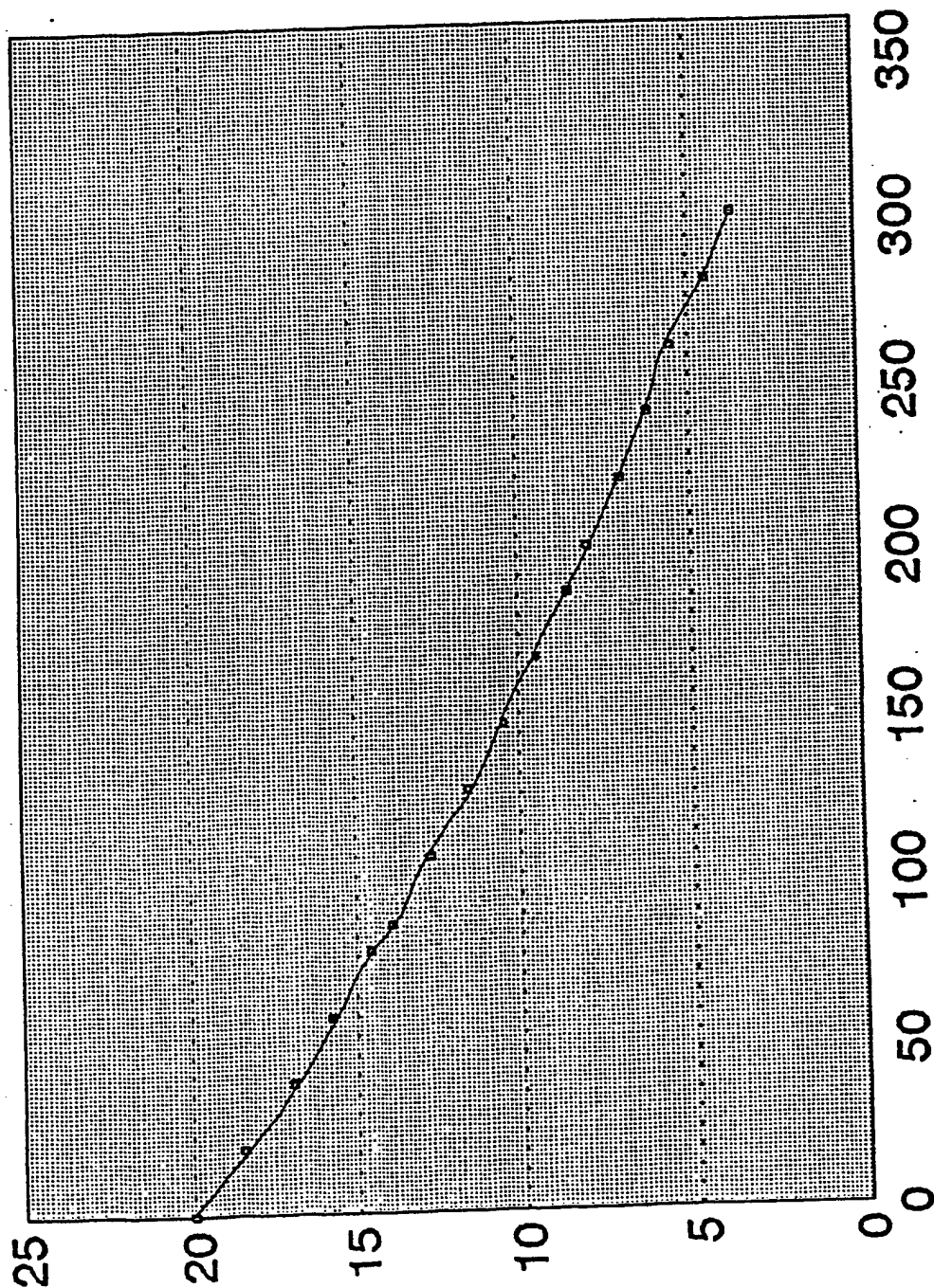
APPENDIX E



EVAPORATION DATA

ZYMARK

APPENDIX E

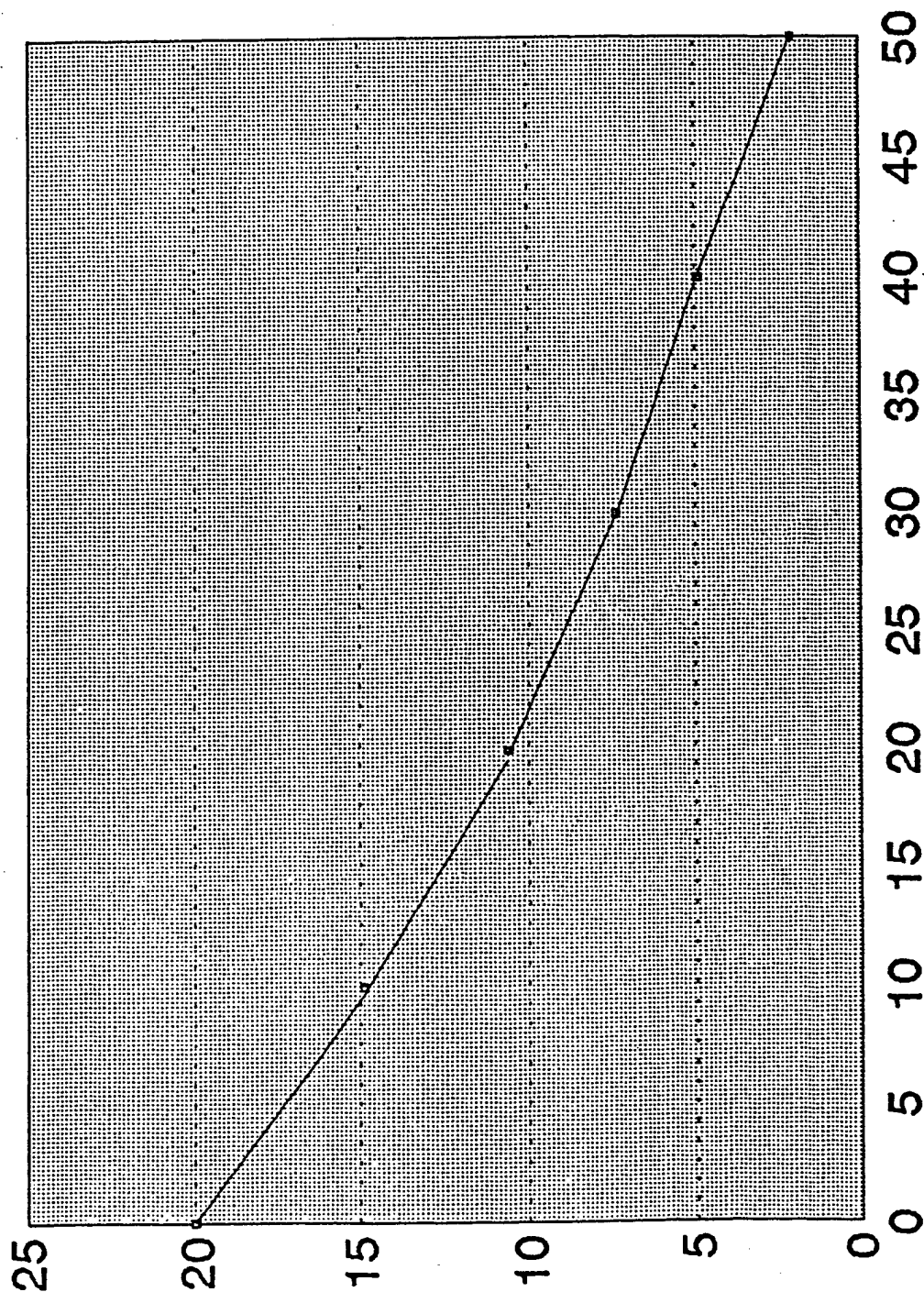


Water

EVAPORATION DATA

ZYMARK

APPENDIX E



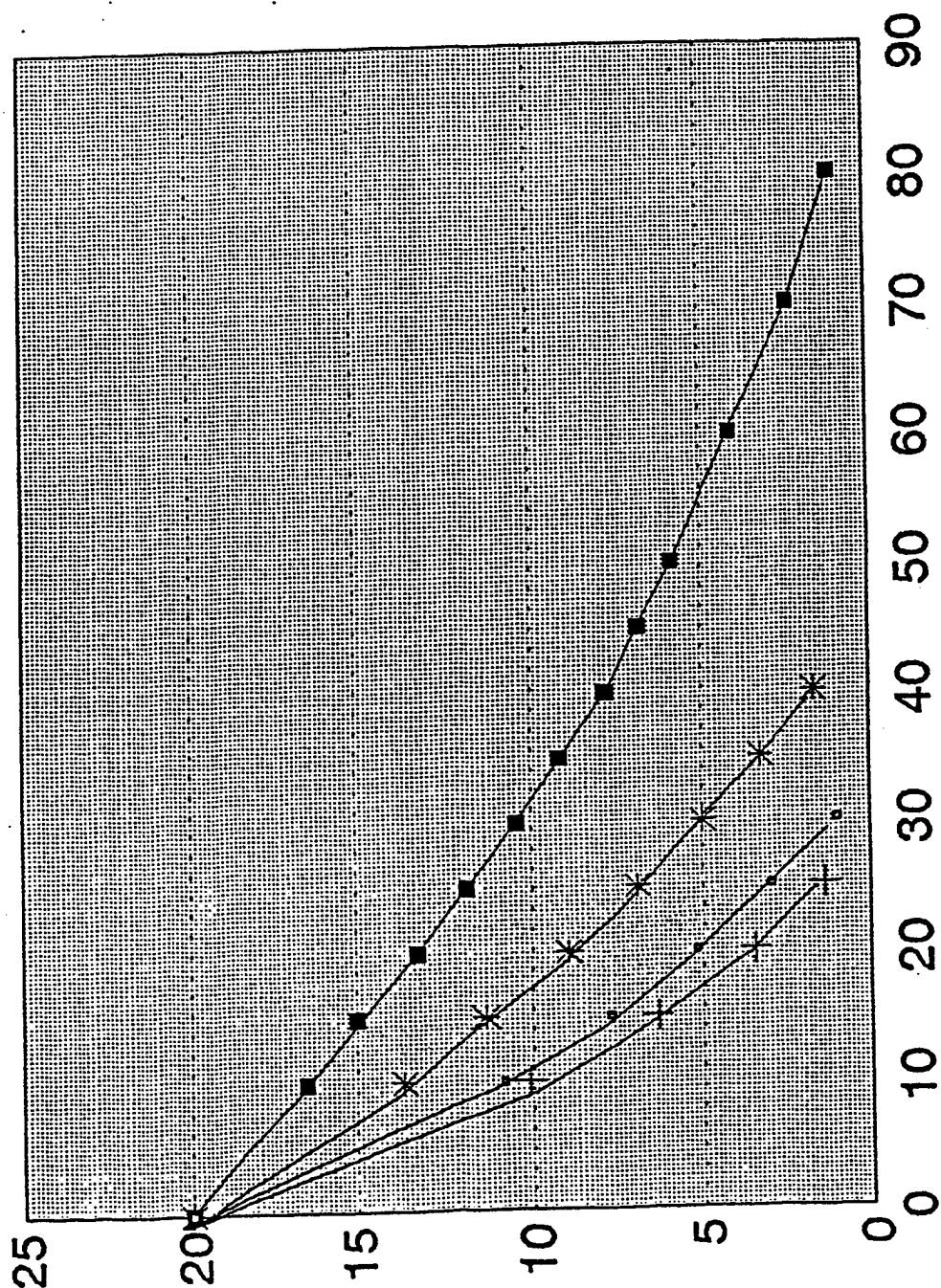
▪ MeOH

50 DEGREES @ 7 PSI

EVAPORATION DATA

ZYMARK

APPENDIX E



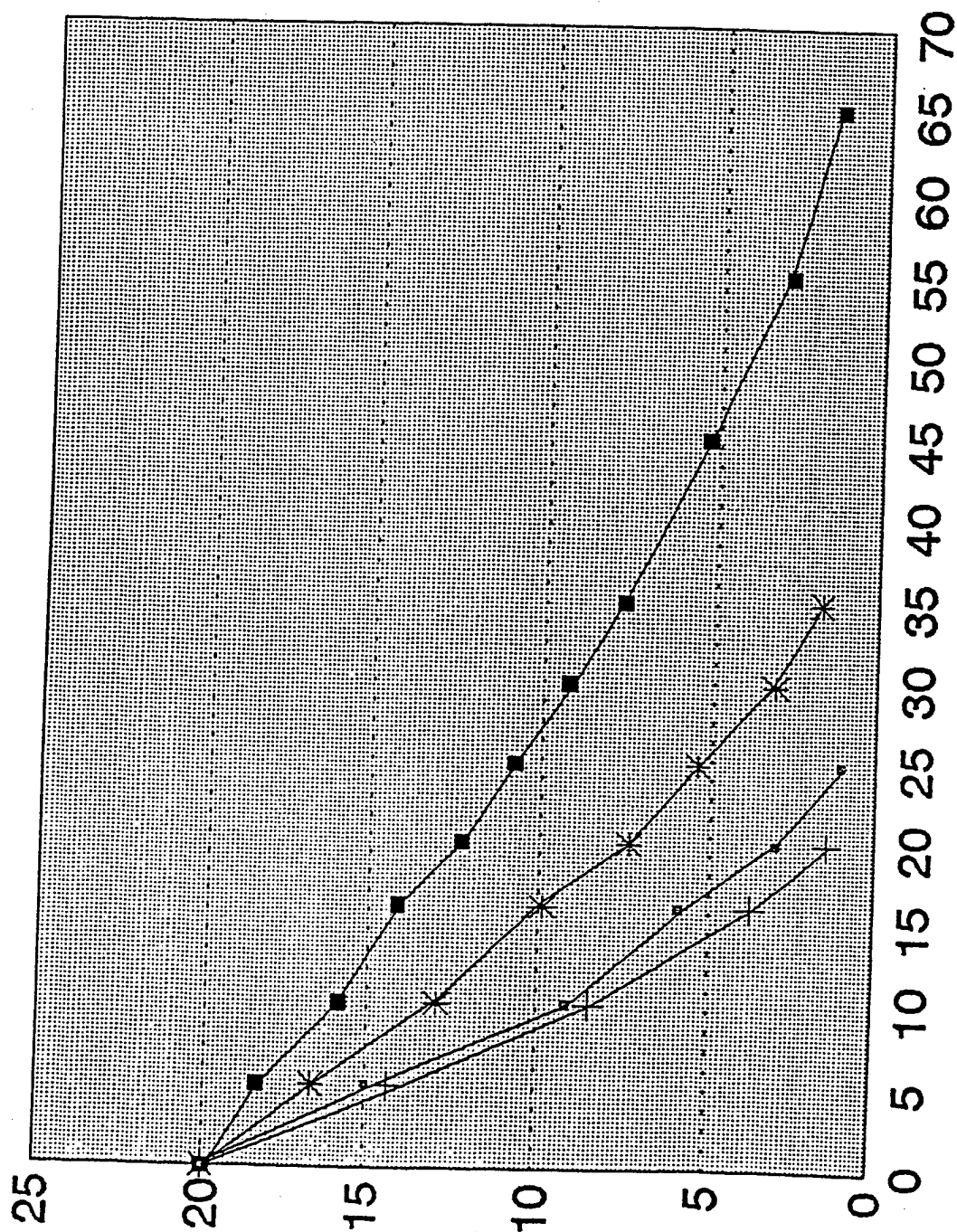
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40 DEGREES @ 7 PSI

EVAPORATION DATA

ZYMARK

APPENDIX E

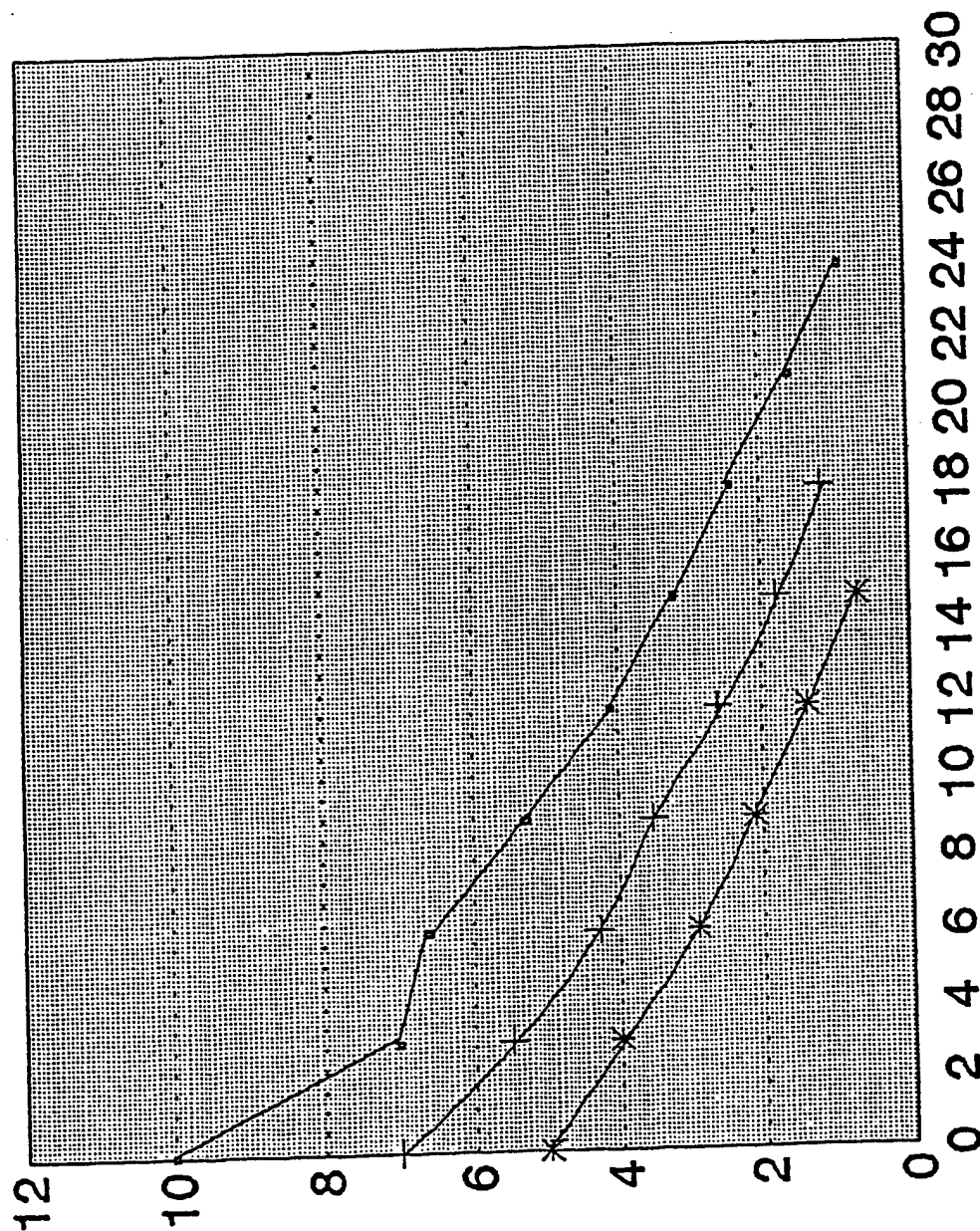


45 DEGREES @ 7 PSI

EVAPORATION DATA

ZYMARK

APPENDIX E



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APPENDIX F

Zymark TurboVap Evaporation Chart - Methanol/Matrix

ZYMARK EVAPORATION CURVE FOR MeOH EXTRACTS (T = 40 °C)

